THE AMERICAN MINERALOGIST, VOL. 48, NOVEMBER-DECEMBER, 1963

EXTERNALLY HEATED COLD-SEAL PRESSURE VESSELS FOR USE TO 10,000 BARS AND 750° C.[1]

W. C. LUTH AND O. F. TUTTLE, *Department of Geochemistry and Mineralogy, The Pennsylvania State University, University Park, Pennsylvania.*

The cold-seal pressure vessel described by Tuttle (1949) has been modified for use at higher pressures and temperatures. This is made possible by the development of new high-strength alloys and by slight changes in the design. The pressure vessel has been used at 750° C. at an argon pressure of 11,000 bars. The alloy, Rene 41[2], has an ultimate tensile strength of approximately 206,000 psi. at room temperature, and 165,000 psi. at a temperature of 750° C. in the heat treated condition. The heat treatment of this alloy, as recommended by the Haynes Stellite Company, is as follows: a solution treat at 1975° F., water quench; a solution treat at 1950° F. for 30 minutes, air cool; age at 1400° F. for 16 hours, air cool.

The pressure vessel[3] (Fig. 1A) is 8 inches in length and $1\frac{1}{4}$ inches in diameter with a $\frac{1}{4}$ inch diameter axial hole drilled in one end to a depth of $7\frac{1}{4}$ inches. To prevent concentration of stress at the end of the $\frac{1}{4}$ inch hole, it is machined as nearly hemispherical as possible. Temperatures are measured in an external well drilled from the closed end of the pressure vessel to 1 inch depth midway between the $\frac{1}{4}$ inch hole and the outside of the pressure vessel. The vessel is threaded as shown in Fig. 1, and to facilitate removal of the closure nut, flats are machined on the pressure vessel near the end which is in the region not excessively heated during the runs.

The alloy can be machined using carbide-tipped tool bits. All machining of this alloy is done before heat treatment.

The closure of the pressure-vessel is made by a cone-in-cone seal as in the original design, but at the higher pressures the steel used for the cone must be a hardenable variety, such as AISI 4140 or 4340. The cone-seal should be heat treated to a Rockwell C hardness of 45. A $\frac{1}{16}$ inch hole is bored axially in the steel and a standard $\frac{3}{16}$ inch Harwood[4] fitting as shown in Fig. 1B is machined on the outer end of the seal. The cone is

[1] Contribution No. 62-120, College of Mineral Industries.

[2] Manufactured and developed by General Electric Co. and manufactured under license by Haynes Stellite Division of the Union Carbide Co.

[3] The pressure vessels are manufactured by Tem-Pres Research, State College, Pennsylvania.

[4] Harwood Engineering Co., Walpole, Mass.

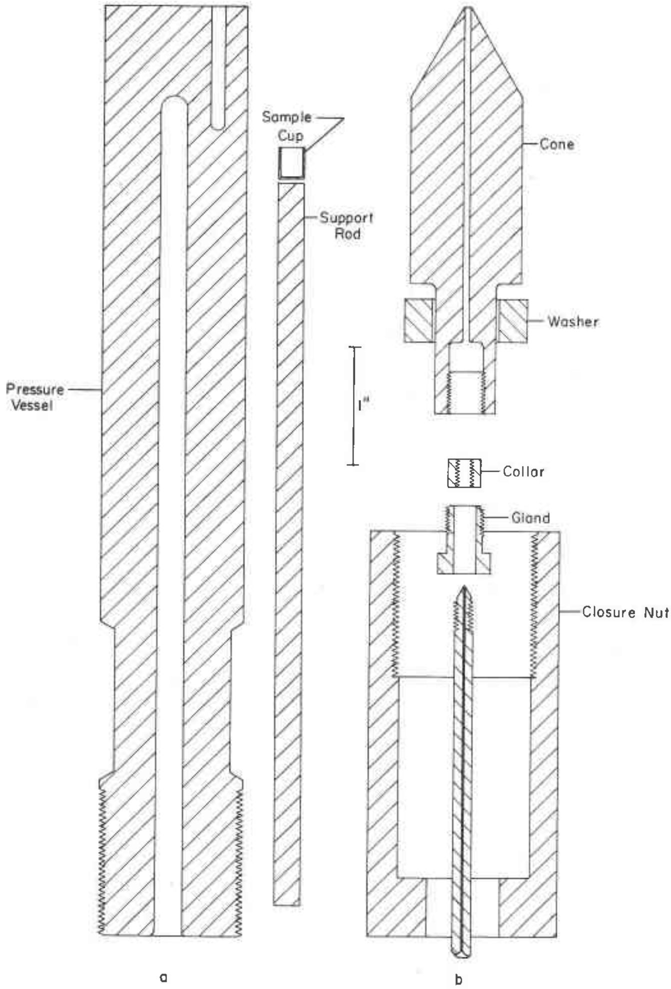

FIG. 1. Cross-section of pressure vessel and components for use to 10,000 bars at 750° C. A. Pressure vessel, B. Closure components.

backed by a 4140 or 4340 steel washer heat treated to Rockwell C50 and lubricated with molybdenum sulfide lubricant. This combination prevents rotation of the cone and consequent galling in its seat when the closure nut is tightened. Pressure supply lines are standard $\frac{3}{16}$ inch tubing supplied by the Harwood Co.

We have found that the closure nut also must be heat treated, and here we suggest AISI 4140 or 4340 heat treated to a Rockwell C hardness of 38 to 40. The threads on the bomb nut should be loose-fitting and well lubricated to prevent galling.

The pressure vessel is mounted in a vertical position with the closure at the bottom, and the samples are placed in a small cup supported by a steel rod. The pressure vessel is heated by a resistance furnace with $1\frac{1}{2}$ inch internal diameter. The furnace is mounted in such a way that it can be removed from the pressure vessel at the end of a run and the vessel can be quenched by directing a stream of air against the hot portion. An annular ring with perforations on the inside is supported at the bottom of the furnace and automatically comes into the correct position for quenching when the furnace is raised from the pressure vessel. Raising the furnace and turning on the air for quenching is carried out by remote control.

Temperature is measured by chromel-alumel thermocouples, one placed in the well in the pressure vessel, and a second outside the well just above the bomb for controlling the temperature of the furnace. Pressure is measured by a Harwood manganin cell, whose output is measured and continuously recorded by a Foxboro Dynalog Recorder. The highest pressure that has been used with this device is 11,000 bars at 750° C. Runs up to one week at 740° C. at 10,000 bars are routine.

Initially, the pressure vessels were machined from 1 inch stock,but after several runs in the range 700–750° C., a number of them failed. We have had no failures with the $1\frac{1}{4}$ inch pressure vessels.

The principal advantage of this type of apparatus is the low cost of operation and the relative simplicity. One can operate this pressure vessel, or a series of these pressure vessels, at a fraction of the cost of internally heated pressure vessels.

THE AMERICAN MINERALOGIST, VOL. 48, NOVEMBER-DECEMBER, 1963

THE GROOVED PLATE, A SIMPLE PETROGRAPHIC AID FOR SIZE MEASUREMENTS OF ELONGATE MINERALS[1]

K. A. SARGENT, *U. S. Geological Survey, Federal Center, Denver, Colorado.*

INTRODUCTION

The increasing use of data derived from measurements of elongate grains, such as zircon crystals, has created a need for techniques that permit the rapid measurement of a statistically significant number of grains. By use of the method to be described, wherein the grains are first aligned by means of a grooved plexiglass plate and then measured with

[1] Publication authorized by the Director, U. S. Geological Survey.